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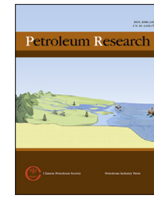

Study the suitability of neem seed oil for formulation of eco-friendly oil based drilling fluid

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A B S T R A C T

The present work intended to develop an invert emulsion drilling mud from biodegradable neem seed oil as an alternative to conventional diesel-based drilling mud. This would present comparable properties and performances with API standard requirements, ecologically friendly and at a low cost. Biodiesel produced from non-edible neem oil was used as the continuous phase in an invert emulsion-based drilling mud. Different tests to investigate the applicability of the mud for drilling applications were conducted. The rheological and lubricity tests results indicate that the neem oil biodiesel-based drilling muds are comparable with conventional diesel-based drilling muds. The produced biodiesel shows a significantly better flash point of 168 °C than the traditional diesel flash point of 70 °C; indicating better fire safety than the conventional diesel. The lubricity and rheological data indicate the formulated mud with neem seed oil biodiesel fair reasonably well with conventional diesel and within the API requirements. In general, the preliminary results show that neem oil biodiesel is a potential alternative to conventional organic oil in formulating oil-based drilling mud in terms of technical and environmental analysis.

1. Introduction

The quest for sustainable development owing to increasing environmental challenges has increased the interest in searching for renewable, biodegradable, and environmentally friendly products and process developments (Akubude and Nwaigwe, 2016; Anya et al., 2012; Demirbas, 2008; Hasan and Rahman, 2017; Sui et al., 2021). The demand for energy has increased massively over the years due to the ever-increasing rate of industrialization, modernization and advancement. The resultant impact of this is the search for hydrocarbon and drilling of oil wells in deeper formations, unexplored regions and hostile environments. Drilling fluids are functional fluids used to perform several functions during drilling operations. It consists of both the continuous phase and the dispersed solutes. iIts major functions include: lubricating and cooling of the drill bits, transportation of the cuttings to the surface, hole stability control and balancing the formation and wellbore pressures (Ali et al., 1987; Fadairo et al., 2012; Fadairo et al., 2012; Fadairo et al., 2016; Fadairo et al., 2017; Karakosta et al., 2021). From the inception of oil well drilling, there have been two major types of drilling fluids (Invert emulsion- and water-based muds, IEDMs & WBMs), based on what fluid is the continuous phase. Invert emulsion-based muds comprise of oil-based mud and synthetic-based muds. Initially, water-based muds received general acceptance due to their superior lower environmental effect and low cost of operation. However, drilling through problematic formation (Gambo shale), deep offshore, and in long extended-reach well could prove problematic for water-based muds (Fadairo et al., 2012; Fechhelm et al., 1999; Giwa and Ogunbona, 2014; Boyou et al., 2019). Therefore, in rescuing these situations, operators rely on the use of invert emulsion-based mud, especially the synthetic and the oil-based mud using mineral oil or organic oil, owing to their superior lubricating effect, lower drag and torque, improved well-bore stability during drilling and overall improved drilling

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Nomenclature

NSOME –	Neem seed oil methyl ester
IEDM	Invert emulsion drilling mud
OBM –	Oil Base Mud
WBM –	Water Base Mud
ASTM –	American Society for Testing and Materials
API –	American Petroleum Institute
SBM –	Synthetic Base Mud
LTMOs –	Low Toxic Mineral Oils
NABF –	Non-Aqueous Base Fluids
EPA –	Environmental Protection Agency
cST	Centistokes
cP	Centipoise
ml	Milliliters
RPM –	Revolution per minute
PV –	Plastic Viscosity
YP –	Yield Point
LSO –	Neem seed oil

efficiency. Synthetic-based fluids (SBFs) were first used in the Norwegian Sector of the North Sea to drill in 1990. In the UK sector of the North Sea, SBF was firstly used in 1991, and in the Gulf of Mexico, it was firstly used in 1992. Toward the end of September 1994, 169 wells have been penetrated utilizing engineered based liquids in the North Ocean. In 1995, 49 wells were bored with engineered based liquids in the Norwegian part of the North Ocean, bringing about the release to the sea of an expected 5500 metric tonnes of huge amounts of drilling cuttings. Despite all the numerous advantages of invert emulsion muds, its use has been cut short owing to the negative environmental impacts of its effluents, its degree of toxicity, and non-biodegradable nature (Fadairo et al., 2012; Ismail et al., 2015; Li et al., 2021). Numerous research works have been reported on the development of invert emulsion-based muds formulated with biodegradable oil as the continuous phase (Curbelo et al., 2021). Fadairo, Adeyemi, Ameleko and Falode (2012) formulated different seed oil-based muds using Jatropha oil and Canola oil. The rheological properties of both muds were compared with emulsion-based mud formulated with diesel at varying temperature using an artificial neural network (ANN). They reported environmentally safe drilling muds with properties in tandem with those observed with diesel-based mud. Nonetheless, many seed oils used recently in formulating emulsion-based muds may have compared favorably with mineral oil or organic-based mud in terms of performance but their use has been limited because most of the oil is edible and will naturally cause artificial scarcity in their supply if they were to be deployed commercially (Hasan and Rahman, 2017; Fadairo et al., 2012; Fadairo et al., 2016; Fadairo et al., 2017). Several drilling challenges have been reported for both non-edible and edible seed oil-based emulsion muds. These range from congealing of the oil during cold flow which could impact the mud viscosity, poor thermal stability at elevated temperature, and unstable wellbore conditions during drilling in challenging formation. Additionally, most seed oil-based emulsion mud is prone to hydrolysis in basic or acidic medium (Oseh et al., 2019: Oseh et al., 2019). Therefore, the research focus is on developing high performance and operationally stable lubricants that could replace the aqueous phase in emulsion-based drilling mud for improved drilling process. The recent trend in formulating invert emulsion muds today is the use of biodiesel derived from seed oils, most especially non-edible seed oils. Generally, biodiesels are products of starches, non-edible plants, microbes, algae, and edible sugars (Giwa, Ogunbona, 2014; Moser, 2009; Nanthagopal et al., 2019), most of which are naturally non-toxic and biodegradable. Biodiesels are simply mono-alkyl esters of long-chain fatty acids that are found in oils and or fats from animal and plant sources (Nanthagopal et al., 2019; Moser, 2009; Oseh et al., 2019: Oseh et al., 2019; Sulaimon et al., 2017). Biodiesel has been reported to boast remarkably better physicochemical properties than conventional organic diesel with higher flash point, low coefficient of friction, better lubricity coefficient, and better electrical stability. This study reports the results of the use of neem seed oil as the functional external phase of biodiesel based-drilling mud in place of the conventional organic diesel. Neem oil is a vegetable oil pressed from the fruits and seeds of the neem (Azadirachta indica), an evergreen tree which is endemic to the Indian subcontinent. It is utilized for natural cultivating and prescription (Karmakar et al., 2012). Neem oil shifts in shading. It tends to be brilliant yellow, yellowish darker, rosy dark-colored, dull dark-colored, greenish darker or splendid red. It has a solid scent that is said to join the smells of shelled nut and garlic (Karmakar et al., 2012). It is made for the most part out of triglycerides and contains numerous triterpenoid mixes that are in charge of the severe taste. It is hydrophobic in nature and serves as an emulsifier in water for application purposes. In this study, neem seed oil biodiesel was used as the continuous phase in formulating a biodiesel based drilling mud for application in drilling operations. The resultant mud system was characterized and investigated for its use in drilling applications. Besides, its rheological and lubricity characteristics were compared with emulsion-based mud formulated with conventional diesel, a mathematical model that relates that basic rheological properties to temperature were developed.

2. Experimental

2.1. Materials

The base oil (Neem seed oil) was procured from a local market, ketu, Nigeria. Analytical grade chemicals, such as toluene, sodium hydroxide (NaOH), potassium hydroxide (KOH), methanol and ethanol were gotten from the chemistry laboratory within Covenant University, Ota, Nigeria. Conventional diesel of grade II standard used for comparison purposes and in formulating the base case of diesel oil-based mud was bought from a local dispensing station (Mobil Oil) in Ota, Nigeria. The conventional diesel also conforms with the American Society for Testing Materials (ASTM) Standards for a grade II diesel (ASTM D975, Grade II-DS500).

2.2. Method

2.2.1. Free fatty acids (FFAs) determination

From the titration method, the conversion rate of FFAs in the esterification with H_2SO_4- catalyst reaction was assessed (Oseh et al., 2019). A solution of 2 g of neem seed oil (NSO) and 10 ml of ethanol was prepared in a beaker by first adding the NSO into the beaker and then adding the ethanol into it also. To start up the titration, 3 drops of phenolphthalein indicator was added into the prepared solution. Another solution of potassium hydroxide (KOH) –in- ethanol (ETOH) was added into the burette dropwise. The solution was agitated continuously during the titration. As the colour of the solution began to change from colorless to pink, the volume of KOH was being monitored. Before the esterification treatment, the FFA content of the seed oil was 4.90% and also the acid value was 10.2 mg KOH/g. Using a two-step transesterification method was used to further analyze the neem seed oil methyl ester (NSOME).

2.2.2. Esterification of neem seed oil

According to previous research Oseh et al. (2019); Moser (2009); Sulaimon et al. (2017) a two-step transesterification method was used to produce the biodiesel. Due to saponification associated with alkaline-catalyzed transesterification and slow reaction time which are commonly associated with this reaction, those methods were used. The decision to adopt H_2SO_4 −catalyzed esterification process was made due to the high FFAs content which was 4.90% (>1% w/w) to reduce it to a level below 1% w/w (Anya et al., 2012; Pantoja et al., 2013; Sathya and Manivannan, 2013). This was done because high FFAs content that is above 1% w/w will result in a soap formation which in turn weakens the efficiency of the catalyst, causes gel formations and also leads to an increase in viscosity and may also make the separation of the glycerol difficult.

The esterification process was conducted as follows: firstly, a borosilicate glassware (500 ml beaker) which contained water was heated to 60 °C by using an electric heater. Neem seed oil of 200 ml was added into 250 ml three-necked bottom flask mounted with a reflux condenser. The flask was then placed in the tray on the heater with a magnetic stirrer and a temperature regulator. The reaction was catalyzed using a mixture of 2 g of sulphuric acid H_2SO_4 and 43.96 g of methanol in the three-necked bottom flask. The mixture was heated for 60 min at 60 °C and then transferred into a separating funnel and left to cool and separate into phases. From the segregated layers, the methanol upper brown layer was removed, and the yellowish oil with NSOME lower layer was kept and washed with 1000 ml of water at 60 °C until a resultant pH of 8.18. The oil was then cooled in a separating funnel at room temperature after the water has been successfully removed. After cooling, the magnetic stirrer was employed to stir the oil for 20 min at 100 °C. NSOME was the oil remaining that contained FFA that was reduced through the second step transesterification method which had KOH as its catalyst.

2.2.3. Transesterification process

A measured amount of 180 g of NSOME was added into a three-necked 250 ml bottom flask which has a reflux condenser. Using an electric heater with a magnetic stirrer and temperature regulator, the flask is heated. A mixture of 1.80 g of KOH concentration and 39.57 g methanol was used as the catalyst in the bottom flask. For about 1hr the mixture was stirred vigorously at 350 RPM (revolution per second) at 60 °C (Oseh et al., 2019; Sathya and Manivannan, 2013). After this, the mixture is then cooled for 3 h in a separating funnel. The products of the reaction were separated using a centrifuge to give LSO methyl esters, methanol, and glycerol. The lower layer was glycerol which was removed by gravity leaving the upper layer of LSO methyl and methanol. A further separation of the upper layer to give LSO methyl esters and methanol separately for the analysis of pure LSO methyl esters. A yield of 82.6% of biodiesel was extracted, collected, washed with warm water and stored before characterization and also for mud formulation. A reduction of the FFA content to 0.23%, and the flash point value of 70 °C was observed due to the two-step transesterification method. Three individual tests were carried out to avoid errors and an average of these were taken for the LSO sample for further examination.

2.2.4. Biodiesel characterization

A portion of the produced biodiesel were tested for their physicochemical properties following ASTM and European committee for Standardization (ECN EN) specifications (ASTM D97, 2002; ECN EN14214, 2003; ASTM D6751, 2009; ASTM D6371, 2010).

2.3. Drilling mud formulation

In line with previous researches (Fadairo et al., 2016; Oseh et al., 2019; Dosunmu, 2010), invert emulsion drilling mud was formulated using neem seed oil.

Table 1 presents the components of the drilling mud formulated and their basic functions for both the neem seed oil invert emulsion mud and the conventional diesel oil-based mud.

2.3.1. Rheological and flow properties tests

The rheological properties and flow characteristics of the two invert emulsion drilling muds (IEDMs) were investigated using an OFITE Model 800 viscometer with eight (8) accurately regulated speed and a broad-ranging shear rate from 3 (Gel), 6, 30, 60, 100, 200, 300 and 600 RPM. The calibration of the viscometer was done in accordance to API recommended practice 13B-1 and 13B. The speed of the viscometer in terms of RPM was adjusted using a regulator knob and the deflection readings taken accordingly as shown on the light enabled dial panel. The deflection readings and the RPM values were subsequently used as inputs for the estimate of rheological properties of the drilling muds, such as mud's apparent viscosity, (AV), plastic viscosity (PV), and yield point (YP) at varying temperatures (25, 40, 50, 70, 80 °C) without aging.

2.3.2. Lubricity test

The drag and torque reducing abilities of the two IEDMs in terms of lubricity coefficient were evaluated using OFITE EP (Extreme Pressure) lubricity tester. The torque readings at precisely adjusted resolution per minute were used to estimate the lubricity coefficient of the drilling muds. The lubricity tester was calibrated according to the manufacturer's specification in line with API standard using deionized water at room temperature and the torque reading taken as expressed below.

Deionized water torque reading at 25 °C = 36.7 (the value is within the range for lubricity of standard deionized water).

3. Results and discussion

Table 2 shows the free fatty acid composition of the neem seed oil, the analysis indicates that palmitic acid and oleic acid are the dominant fatty acids in the seed oil. Table 3 presents the physicochemical properties of the two lubricants used in the formulation of the invert emulsion drilling muds. As observed from Table 3, the neem oil biodiesel exhibits a lower density compared to

Table 1
Formulated Drilling mud composition.

Additive	Function	Units	NSO mud	Diesel OBM
Diesel	Continuous Phase	Ml	245	245
Water	Dispersed Phase	Ml	105	105
Organophilic Clay	Viscosifier	G	5	5
Lime	Neutralizers for fatty acids and alkalinity	G	3	3
Barite	Weight control agent	G	40	40
Ez-mul	Oil emulsifying agent	G	20	20
Invermul	Primary emulsifier	G	18	10

Table 2
Neem oil free fatty acid composition.

Common Name	Acid Name	Chemical Formula	Lipid Number	Composition Range (%)
Omega-6	Linoleic acid	$C_{18}H_{32}O_2$	C18:2	6–16
Omega-9	Oleic acid	$C_{18}H_{34}O_2$	C18:1	25–54
Palmitic acid	Hexadecanoic	$C_{16}H_{32}O_2$	C16:0	16–33
Stearic Acid	Octadecanoic acid	$C_{18}H_{36}O_2$	C18:0	9–24
Omega-3	Alpha-linolenic acid	$C_{18}H_{30}O_2$	C18:3	–
Palmitoleic acid	9-Hexadecenoic acid	$C_{16}H_{32}O_2$	C16:	–

Table 3
Physicochemical Properties of commercial grade 2 diesel type and neem oil biodiesel.

Properties	Neem Biodiesel	Diesel #2	ASTM D6751-02 Specification
Density (gm/ml)	0.808	0.850	0.875–0.9
Viscosty (cP)	5.76	2.6	1.9–6.0
Flash Point (°C)	168	70	>130
pH	8.18	10.68	N/A

conventional diesel but generally falls within the ASTM D6751-02 specified standard. The reduced density observed in the biodiesel could be explained as due to the two-stage esterification that resulted into removal of a large fraction of the fatty acid content, free and bound glycerin contents initially in the raw neem seed oil before conversion to biodiesel (Demirbas, 2008). High flash point indicates better fire safety, transportation and storage (Oseh et al., 2019a-b). The neem seed oil biodiesel exhibits higher flash point than the conventional diesel which implies the produced biodiesel will demonstrate better fire safety and storage. The produced biodiesel shows comparatively higher viscosity than the conventional diesel, which can be attributed to the large fraction of monounsaturated and total saturated fatty acids in the biodiesel (Oseh et al., 2019; Li, Zhao, 2018). Figs. 4 and 5 present the API allowable specifications for the rheological properties and lubricity coefficient of standard oil and IEDMs. These specifications were used as basics for the evaluation of the two formulated mud's performances.

3.1. *Rheological properties of drilling muds*

The experimental results show the performance of the neem oil biodiesel-based drilling mud and conventional diesel drilling mud clearly indicate the suitability of the neem oil biodiesel-based drilling as an invert emulsion drilling fluid and also compares well with the conventional diesel-based mud (Table 4). From Fig. 1, it can be deduced that the shear stress/rate relationship for the conventional diesel-based drilling mud exhibits Bingham plastic model with a constant slope, which is a visco-plastic fluid behavior in which fluids behave as a rigid body at low shear stresses but flows as a viscous fluid at high stresses. The neem oil biodiesel-based drilling mud demonstrates the Herschel-Bulkley fluid behavior which is typical of non-newtonian fluids. In this case, the shear stress/rate relationship is non-linear with a changing slope. In terms of rheological data, there is no significant difference

Table 4
Rheological properties of the formulated muds.

Rheological Properties	Neem bio-diesel	Conventional diesel	API Specifications
Plastic viscosity	15	23.5	<35
Yield point	25	35	15–25
Density (ppg)	8.05	8.1	8.65–9.60

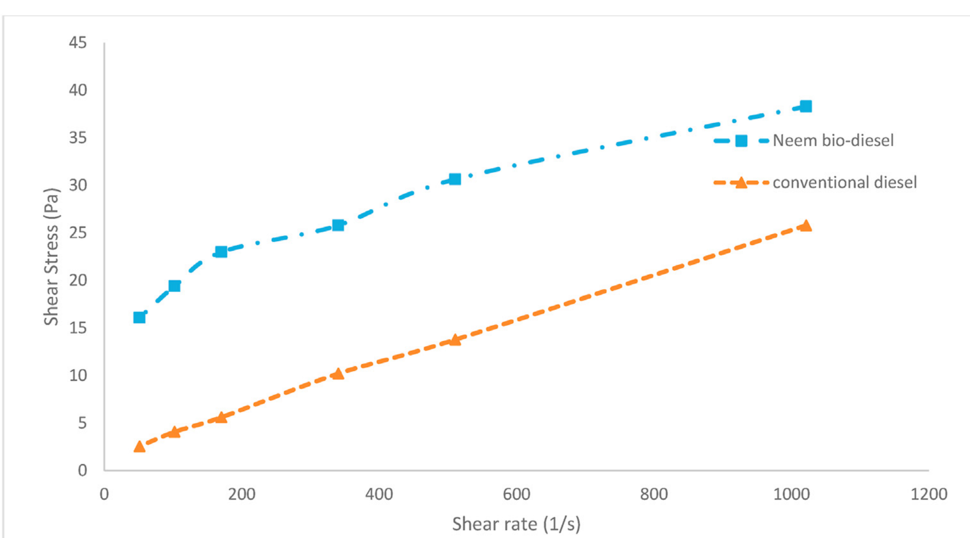

Fig. 1. Shear stress vs shear rate data for invert emulsion drilling mud formulated with neem seed oil biodiesel and conventional diesel.

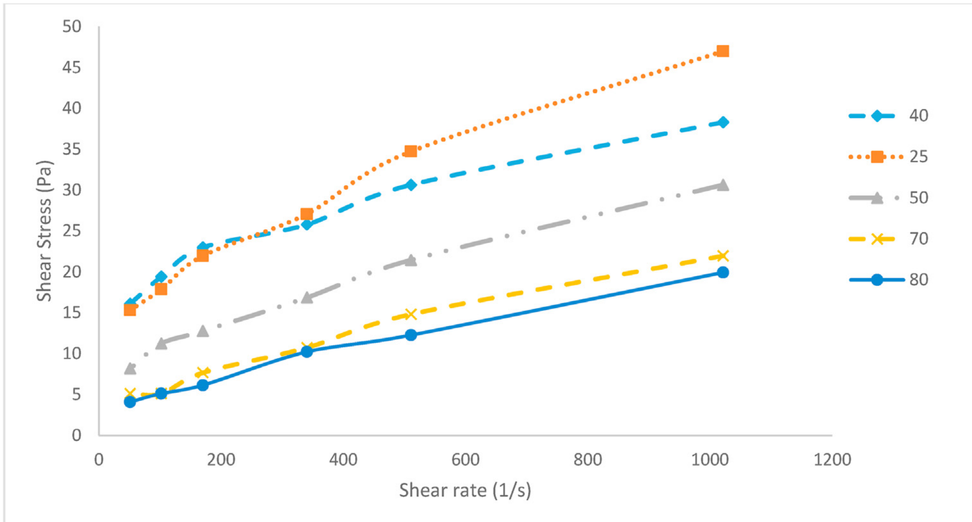

Fig. 2. Shear stress vs shear rate data for invert emulsion drilling mud formulated with neem seed oil biodiesel at varying temperatures (°C).

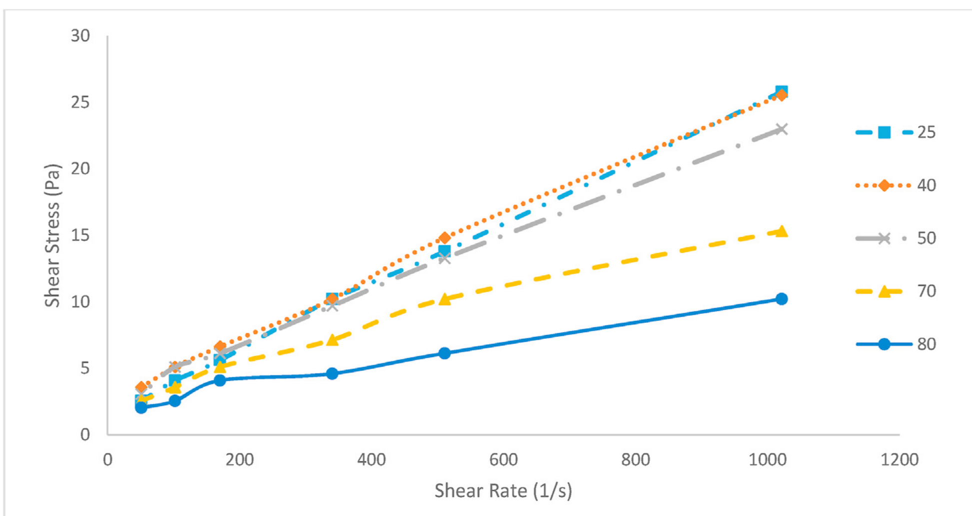

Fig. 3. Shear stress vs shear rate data for invert emulsion drilling mud formulated with conventional diesel at varying temperatures (°C).

between the two formulated muds in laboratory conditions.

The plastic viscosity and yield point of the neem oil biodiesel-based drilling fluid give relatively lower values than the conventional diesel oil-based mud, this could be credited to the existence of fatty acid chain length and double bond of carbons in the neem oil biodiesel-based mud (Oseh et al., 2019). Figs. 2 and 3 present the temperature effects on the two muds which indicates the stability of the two formulated muds. As observed from the plots, both exhibit reduction in the shear stress/rate relationship as the temperature is raised. This behavior can be explained to be due to the energized liquid molecules in the mud moving freely rather than bonded as the temperature is raised. Demirbas (2008) explained this thus; the energized molecules could move freely because as the temperature is raised, the cohesive intermolecular forces within the liquids strongly oppose the energized molecules within the liquid.

3.2. Lubricity results

The effectiveness of any lubricant to reduce the friction between any two metallic surfaces is measured by the lubricity tester and quantified as the coefficient of lubricity. This is a characteristic operators desired in drilling fluids. The coefficient of lubricity for both drilling muds was measured using the Lubricity Tester. The API standard for the coefficient of lubricity for an oil-based mud should be between 0.15 and 0.20. After taking the torque readings at different revolutions per minute, the values of the lubricity coefficient for both the conventional diesel-based drilling mud and the neem oil biodiesel-based drilling mud are as presented in Table 5. Generally, knowledge of the lubricity coefficient is necessary in well path and drill string design, and equally in mud formulation by service companies (Fadairo et al., 2012). It can be observed from Table 5 that invert emulsion mud formulated using neem oil biodiesel exhibits a lower coefficient of friction compared to the conventional diesel-based mud. However, both muds fall within the API specified limits for oil-based mud (Table 6). Therefore, the formulated mud could possibly work where drag and torque reduction is of interest. Significant reduction in lubricity coefficient is equally a function of the mud composition (Maidla and Wojtanowic, 1990), which is the solid suspensions (clay-based

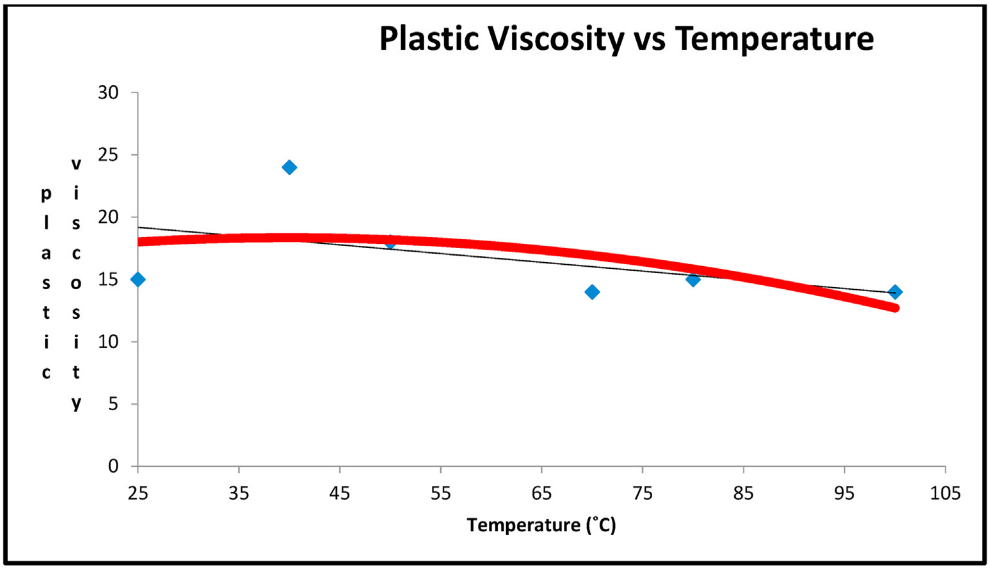

Fig. 4. Polynomial regression curve for the variation of plastic viscosity with temperature.

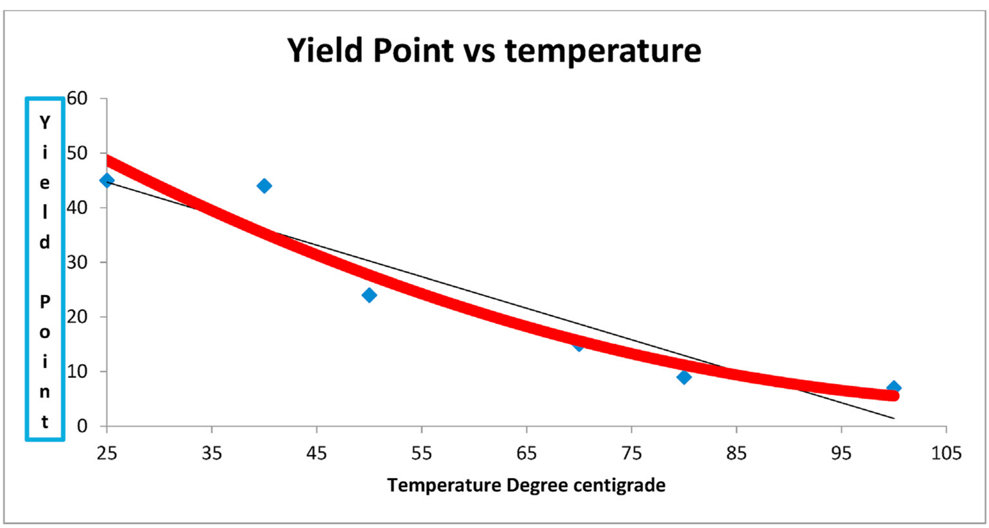

Fig. 5. Polynomial regression curve for the variation of yield point with temperature.

Table 5
Lubricity coefficient for the formulated muds.

RPM values	Lubricity coefficient	
	Neem biodiesel based mud	Conventional diesel mud
60	0.173	0.215
100	0.157	0.185
200	0.192	0.32
300	0.178	0.252
600	0.184	0.2
Lubricity coefficient Average	**0.1768**	**0.2344**

mud systems gives good lubricating performance than non-clay or polymer-based mud systems). The lower coefficient of friction observed in neem oil biodiesel-based drilling mud indicate that it creates a better thin film between the rotating ring and the static test block to give good separation distance and limits the effective contacts at the interface. The small fraction of free fatty content and

Table 6
Lubricity coefficient of both drilling muds compared with standard API.

Standard low	Biodiesel Mud	Conventional Mud	Standard High
0.15	0.18	0.23	0.2

Table 7
Experimental results against model prediction for plastic viscosity and yield point.

Temperature (°C)	Experimental Yield Point (ib/100 ft^2)	Predicted Yield Point (ib/100 ft^2)	Deviation (ib/100 ft^2)	Experimental Plastic Viscosity (Pa.s)	Predicted Plastic Viscosity (Pa.s)	Deviation (Pa.s)
25	45	48.687	**3.687**	15	17.9795	**2.9795**
40	44	35.262	**−8.738**	24	18.269	**−5.731**
50	24	27.637	**3.637**	18	18.062	**0.062**
70	15	15.567	**0.567**	14	16.688	**2.688**
80	9	11.122	**2.122**	15	15.521	**0.521**
100	7	5.412	**−1.588**	14	12.227	**−1.773**

glycerin in the neem oil biodiesel-based mud can be credited for giving gliding slippery surface in the interface between the static block and the rotating ring and thus reduces the coefficient of friction in drilling muds.

3.3. Rheological properties modelling

Accurate estimation and prediction of the rheological behavior of drilling mud are essential for precised evaluation of friction pressure losses in drilled holes during mud circulation, wellbore stability and cutting carrying controls especially as a function of temperature. Regrettably, most existing rheological models such as Powe law, Carson and Herschel-Bulkley and Bingham plastic do not account for the effect of temperature on some of the rheological properties. Plastic viscosity and yield point which are the two important rheological properties responsible for friction loss control and cutting carrying capacity were modeled as a function of temperature from the results of the neem seem biodiesel-based mud rheological behavior experiment. This was achieved using a python programing language and Microsoft Excel, a polynomial regression correlations were obtained for both plastic viscosity and yield point (Eqs. (1) and (2)).

$$PV = -0.0016T^2 + 0.1233T + 15.897 \quad (1)$$

$$YP = 0.0053T^2 - 1.2395T + 76.362 \quad (2)$$

Figs. 4 and 5 present the results of the polynomial regression of the variation in plastic viscosity and yield point with temperature with the limit of the data analyzed. From the result, it can be deduced that the polynomial regression works well with the yield point with R^2 value of 0.960 than the plastic viscosity with R^2 value of 0.8222. Table 7 shows the comparison between the experimental data and the model prediction over the range of temperature analyzed. The results show the model prediction exhibits some level of accuracy at some temperatures. Therefore, it can be used as a guide to what the range of plastic viscosity and yield point would be over that range of temperature.

4. Conclusions

In this current work, the following conclusions were drawn:

i. The neem oil biodiesel produced is environmental friendly and thus the drilling mud formulated with it is expected to have no detrimental effects on the surrounding environments, ecosystem and habitats.
ii. The newly-designed neem oil biodiesel-based drilling mud shows performance similar to the rheological performance of widely used diesel-based drilling mud. However, it shows better lubricity characteristics than the conventional diesel-based drilling fluids.
iii. The good rheological and lubricity, compatibility with OBM additives, Eco friendliness, low cost and general availability make the application of neem oil biodiesel a possible candidate for biodiesel-based drilling muds.

Declaration of competing interest

I write to declare that there is no conflict of interest between the authors of the research paper submitted for publication in your reputable journal.

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